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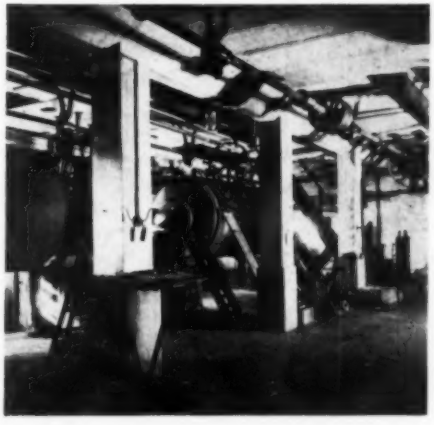

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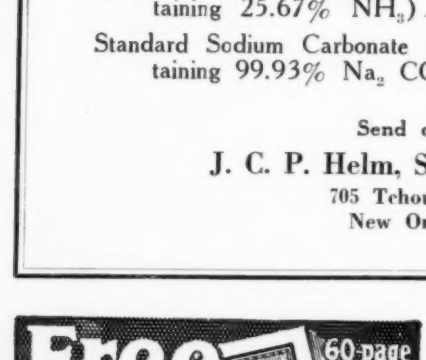

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The Advantages of Purely Absorptive Carbon in Decolorizing

Its Relation to Elimination of Emulsifying Agents

By JOHN P. HARRIS

DECOLORIZING of vegetable oils has long been accomplished by means of Fullers' Earth, and for many years Fullers' Earth constituted the sole purifying and clarifying agent employed, in addition to its decolorizing ability.

Most refinery operatives will tell you of the "Fullers' Earth" flavor which comes to oil when it has been treated with Fullers' Earth. Whereas, actually, Fullers' Earth possesses, and imparts to the oil, no flavor any more than nickel imparts a flavor during hydrogenation. But, in both cases, a peculiar unmistakable odor and flavor is imparted to the oil. This I shall call, for want of a better name, a "catalytic" flavor, because, just as the nickel acts as a catalyst at certain temperatures to cause hydrogen to combine with the oil to convert unsaturated fatty acids, so, I believe, Fullers' Earth acts as a catalyst, at certain definite temperatures, to cause oxygen from the air to combine with oils or fats, whereby a bleaching and whitening occurs.

Many decolorizing agents other than Fullers' Earth have been used, most of them depending on a like catalytically induced oxidation, for a bleaching action. Among these bleaching agents may be mentioned acid treated clays, which for a time proved rather popular; and acid treated carbons, where activation is induced by means of an acid treatment.

So far, a light color is of such importance to the refiner that on

John P. Harris

most oils and fats he is ready to risk a little oxidation in order to achieve a very light product, since as yet no *complete* bleaching agent for cottonseed oil has been commercially prepared which does not tend to oxidize the oil.

Every intelligent refiner knows and deprecates this tendency toward oxidation thus induced, because he realizes that oxidation predisposes an oil to rancidity. I do not mean that bleaching makes an oil rancid, nor that the absorption of a small amount of oxygen makes an oil rancid, but I do mean that a virgin oil, totally without oxygen, may absorb a considerable amount of oxygen while still remaining sweet to taste and smell, but that there is a point of satura-

tion, when an *excess* of oxygen generates rancidity; first, perhaps, marked by a flat or stale flavor; then becoming positive, acrid rancidity. Elimination of this initial oxidation, no matter how slight, would, thus, increase the keeping quality of the oil just that much.

A Purely Absorptive, Non-Catalytic Carbon

While, as stated above, there is no complete bleaching agent for cottonseed oil, which possesses markedly strong decolorizing ability without a catalytic reaction, there is a non-acid, vegetable, activated decolorizing carbon, which produces decolorization by pure absorption. This carbon possesses such a remarkably strong affinity for resins, albuminoids, phosphatides, mucilaginous matter, as well as natural pigmentation, that it cleans, purifies and decolorizes (being especially valuable for the removal of red color), merely by straight absorption, and it does this without exerting any catalytic action whatever upon the oil.

It is interesting to observe that temperature has little effect upon the decolorizing properties of this form of carbon. Catalytic combination occurs between oil and most bleaching agents at temperatures around 180° F., and treatment with such agents at temperatures substantially lower than this would be valueless, yet such substantially lower temperatures produce equally good results with this pure form of carbon, proving that its decolorizing properties do not come, even in part, from catalytic action. Further proof comes from the utter lack of the "catalytic" flavor described above, in fact, this carbon actually does a noticeable job of deodorizing within certain limits, leaving the oil purer and sweeter, as well as whiter, for having been treated with it.

So the wise refiner of cottonseed oil minimizes his chances of oxidation as completely as possible, by using this carbon, and it is noteworthy that this carbon, when used supplementarily to other bleaching agents, actually seems to largely do away with the "catalytic" flavor produced by the other bleaching agents.

A Complete Decolorant for Some Oils

With some oils, such as coconut, corn and peanut, this carbon, in a very small amount, suffices *entirely* for decolorizing, leaving the oil free from oxidation and absolutely clean and pure.

It may be contended from this that the sheer power of absorption which distinguishes this class of carbon from all others, comes close to making it a 100% refining agent. That is, it tends even to neutralize, as well as to decolorize and deodorize. It has been reported by many users that such a carbon actually absorbs some of the free fatty acids as well as the other impurities from the oil, whereas treatment with other decolorizing agents invariably tends to increase the free fatty acid content.

Preparation of Smalley Foundation Samples

THOMAS C. LAW, Atlanta, Georgia, has very kindly consented to prepare the samples for this year's Smalley Foundation Co-operative Checkmeal Analyses of the American Oil Chemists' Society. This work calls for considerable sacrifice on Mr. Law's part and his agreement to undertake the work has earned him the gratitude of the Society.

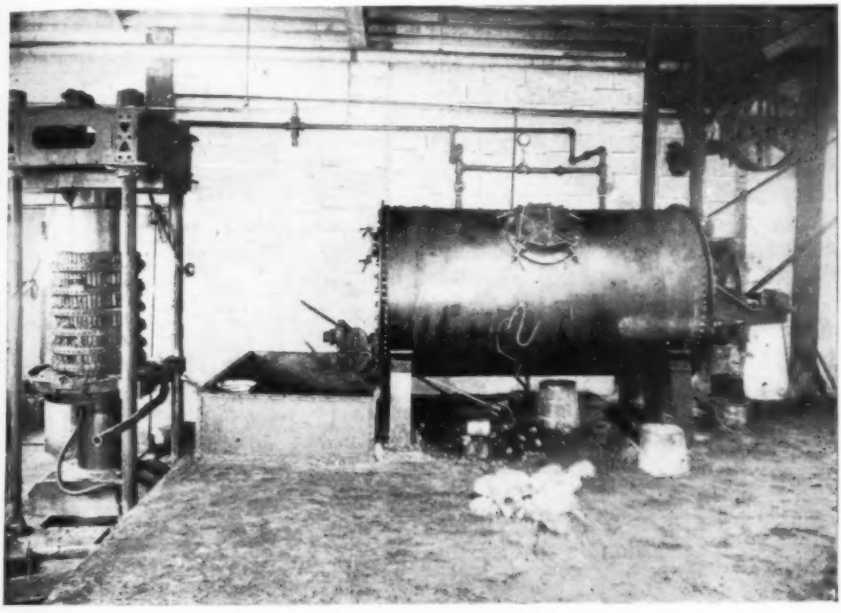
Dry-Rendering of Animal Products

A description of the new process used in the production of edible and inedible fats.

By ROBERT P. BENNETT

ANIMAL fats as they occur in the adipose tissues of the living animal are composed in the main of stearin, palmitin and olein in varying proportions. Chemically fats are neutral substances composed of carbon, hydrogen and oxygen.

If we could process these fats so as to preserve their natural fine qualities intact, we would confer an inestimable boon upon mankind. We are always striving toward this but never quite attaining it.



Dry-rendering plant in operation

Edible Fats

Fats, as they are taken from the freshly killed animal are neutral in flavor, odorless and contain no free fatty acid. They are white except for the straw color of certain beef fats, and are wholesome and sweet.

As these fats occur in the animal tissues they are closely associated with the proteins or essential constituents of all living cells so that to recover them it is necessary to separate them from these proteins and other substances. Since these proteins are of great food value it is equally necessary to recover them in as pure and wholesome condition, as possible.

Therefore, a process of separation with preservation is indicated and one such process is rendering —or the application of sufficient heat to effect such separation.

Unfortunately, the moment that life is extinct in the animal, bacterial action begins, which immediately operates on the fats to separate the glycerine from the fatty acids and produce free fatty acids. Free fatty acids never remain constant. They are perpetually increasing. When in the rendering process heat is applied, particularly in the presence of moisture, the free fatty acid content increases rapidly.

Again, the fat structure is delicate and easily broken down and when fats are rendered at high temperatures in the presence of water a chemical process of decomposition sets in known as hydrolysis.

Thus fats rendered by live steam introduced into them deteriorate to such an extent that perfectly neutral fats fresh from the killing floor will develop from ½ to 1½% of free fatty acid during the process of cooking by such a method.

Furthermore, fats have a great affinity for oxygen at high temperatures and oxidation is a forerunner of rancidity. The keeping qualities of fats, the flavor and the color are directly dependent upon the degree of oxidation.

When therefore, we are dealing with edible fats the rendering process employed must take into account all of the adverse factors above considered.

If the process employed will not deliver products low in acid, of good color, taste and keeping qualities, all within definite close limits, then our fresh fats have passed out of the edible class into the inedible.

Inedible Fats

When we come to inedible fats, we meet an entirely different set of conditions. The tallow or grease rendered from these is largely used in the soap industry and the solids or proteins for stock or chicken feed.

The raw fats entering into inedible come from a great variety of sources; from butcher shops, packing houses, dead animals, hotels, restaurants, etc., and since they are never strictly fresh the acid content will vary from 2 to 50%. It is customary therefore, to segregate these fats and render them to a considerable extent on the basis of a general average acid content. Fresh shop fats for example are rendered to produce a high grade inedible tallow running 4 to 5% F.F.A. Hotel greases are handled separately to produce a different grade of product of higher acid content. Each grade has its limits of acids, color and titre as related to market price; raw materials which might produce a low acid tallow can easily be mishandled and produce a lower grade product than would be the case where they properly processed.

Process Requisites

It is apparent therefore that certain general principles underlie any successful rendering process.

Briefly they are as follows:

1. The raw stock must be processed at the earliest possible moment after receipt of the raw material, to avoid any further increase in free fatty acid.
2. The raw materials should be processed in the shortest possible time—for prolonged exposure to heat, particularly in the presence of moisture, increases the fatty acid content.

3. The raw materials should be rendered at as low a temperature as possible to avoid increase in F.F.A., to prevent oxidation and to keep the oils from discoloration.
4. The raw materials should be rendered without admission of air.

Direct Steam Rendering

In order to appreciate the great advance in rendering processes exemplified by the dry-rendering system we must describe briefly the older system in use for so many years known as the direct steam system.

In this system fat, bones, carcasses or other raw materials were loaded into a vertical steel digester or rendering tank and live steam under 30 to 70 lbs. pressure was introduced directly into the material for from six to ten hours.

Then the content was allowed to settle for an hour, which resulted in three layers of material within the tank: at the top a layer of tallow or grease, next a layer of tankwater, then a layer of solids or wet tankage.

The tallow or grease was then drawn off as free from tankwater as possible. The tankwater was next run to vats for skimming such grease as was not drawn off in the first instance.

The solid matter in the bottom of the tank was then dropped out, pressed in a hydraulic tankage press and dried in a steam jacketed or direct heat dryer.

The tankwater, containing from 4 to 6% protein solids in solution and suspension was evaporated in vacuum pans to about 26° Bé and either dried on a steam heated stick roll into concentrated tankage or mixed with the pressed tankage and the two dried out together in the tankage drier above mentioned. The cycle of this process was from 16 to 24 hours. In other words, cooking with live steam added water to the material and the rest of the process was devoted to getting that water out again. Diagram 1 below shows the products of this system and diagram II the equipment required.

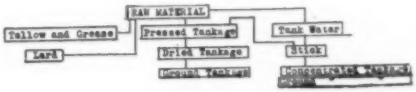


Diagram I

From the foregoing, it will be evident that many secondary products occur in the direct-steam rendering requiring a multiplicity of equipment.

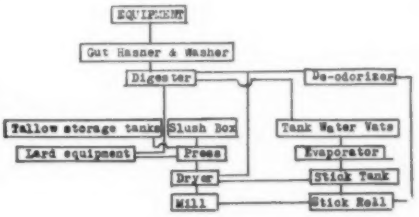


Diagram II

The raw materials are not processed in the shortest possible time; they are not rendered at the lowest temperatures possible. They are rendered in the presence of moisture and under conditions favorable to oxidation. The value of the resultant products does not equal the value of the products of the dry-rendering system, but the cost of production is greatly in excess of the costs under the dry-rendering system.

The Dry-Rendering System

In this system cooking is carried on in steam jacketed melters and no live steam comes in direct con-

tact with the material. The resultant products and the main items of equipment necessary to produce them are shown below in Diagrams III and IV respectively.

Preparation of Raw Materials

When entrails, pecks, paunches, etc., are to be rendered it is first

Diagram III

necessary to hash and wash them in order that the contents, consisting of food in process of digestion be removed.

If this is not done the tallow or grease extracted will have a greenish tinge—be high in free fatty acids and M.I.U., that is, moisture, impurities and unsaponifiable matter taken as a unit and will suffer a material reduction in market value.

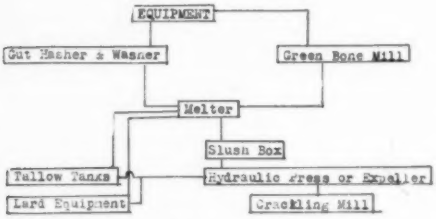
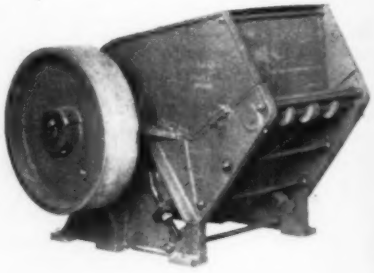


Diagram IV

To prepare offal for the melter a combined hasher and washer is employed. This machine has a set of circular saw tooth disks, rotating at different speeds. These tear and rend the material, automatically passing it into a rotating sloping perforated cylindrical metal screen. As it passes through this screen it is sprayed with water from a perforated pipe, so that the contents of the offal are washed through the screen perforations and the material to be rendered discharged in a clean condition at the end of the washer.

Bones—Carcass Meat

To prepare bones or carcass meat for the melter, a green bone mill or hog is employed. These machines are built in a variety of types and styles and most of them will take the raw material as rapidly as it can be fed and break it down into small pieces.



Green Bone Mill

Milling of the green bones and carcasses is one of the most important and necessary steps in the dry-melting system. We have already stated that one of the fundamentals of efficient rendering is rapidity of operation. If whole bones and large pieces of carcass meat were placed in the melter, a great increase in time of cooking would result, for the reason that the necessary heat could not penetrate such pieces rapidly and disintegration would be slow, thus subjecting the balance of the charge to excessive or over-cooking with resultant detriment to the tallow. The cooking would be uneven and the extraction of the fats incomplete as compared with

extraction from ground material. Furthermore, large pieces of bone prevent satisfactory operation of either hydraulic press or expeller, with the result that the crackling cakes would show great irregularity in fat analysis.

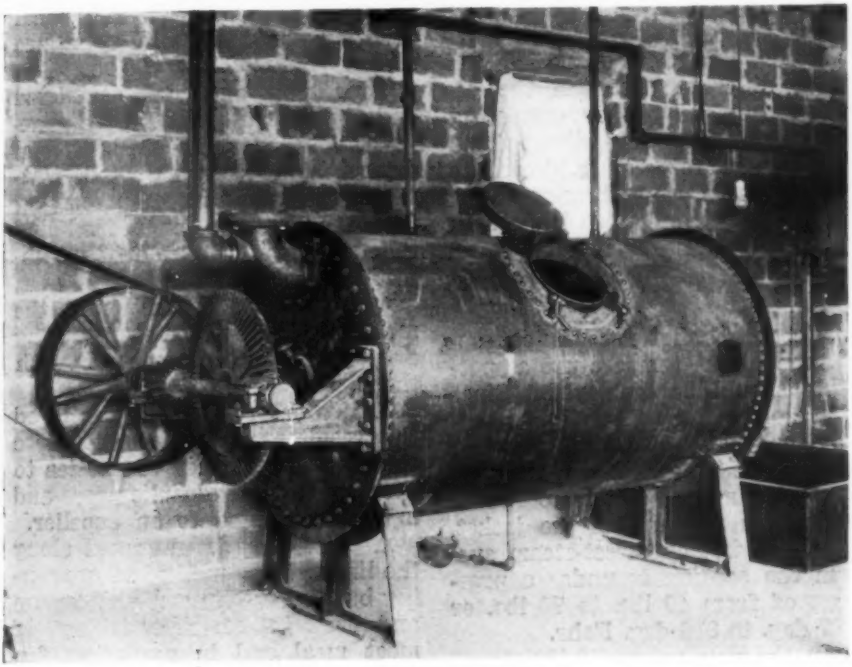
Fats

In the dry-rendering of hog fats for production of lard or in handling shop fats for production of tallow the bulk of a charge to the melter will be fats in pieces which do not require cutting or disintegration as this is accomplished in the melter. Where kidney or cod fats, leaf lard, etc., are received in large pieces they should be cut up, but in general the bulk of such fats needs no pre-treatment.

Melter

The melter is, of course, the heart of the dry-rendering process and upon its construction and method of operation, depend in a large measure the successful results of the process.

The accompanying cut shows the general construction of an efficient melter.



Melter for dry-rendering

The inner shell is built in one piece of heavy steel and there are no staybolts between the shells.

This construction insures a smooth polished inner shell to which no material can adhere and bake. Furthermore, the absence of staybolts means that no steam can leak into the material.

The large vapor outlet permits the immediate escape of the cook-

ing vapors in great volume at low velocity—thus preventing entrainment of fats.

Lead gaskets are employed on loading discharge and testing doors so that no air can enter the melter, thus preventing oxidation.

This melter will cook a charge of straight fat in about 45 minutes, and bone in 1½ to 2 hours. Allowing 40 minutes for pressing a charge, it will be seen that the cycle of operation is approximately 2 hours.

Here we begin to appreciate the great difference between the direct steam and the dry-rendering methods. In the direct-steam method the fat was subjected to high temperature and moisture for 6 to 10 hours and often longer. In the dry-system 45 minutes to 2 hours.

Temperature

We stated that for efficient operation as low temperature as possible should be employed. Since the melter is vacuum-tight it is obvious that a vacuum pump or barometric condenser can be used, thus making it possible to reduce the steam pressure in the jacket.

In the wet or direct-steam system the cooking is under a pressure of from 40 lbs. to 70 lbs. or 287 deg. to 316 deg. Fahr.

In the dry-system 40 lbs. to 60 lbs. of steam is carried in the jacket and so long as any appreciable amount of moisture remains in the material, i.e., the inherent moisture in the tissues—the temperature will remain at about 212° Fahr. Only when the moisture has been driven off will the temperature start to rise—and when it does start to do so—the cooking is completed.

Variant

Occasionally a condition arises where it is inconvenient to install a bone mill or hog. In such cases the whole bone or large pieces of carcass meat may be placed in the melter without milling. A shut-off valve is introduced into the vapor exhaust line and closed sufficiently to permit the vapor driven off of the cooking material to build up a pressure within the tank. In other words a modification of the wet or direct steam process is effected and without additional moisture the bones and meat are disintegrated. This requires three to four hours and when completed and the pressure relieved, the exhaust valve is opened and the real dry-rendering system started. Naturally the total time of processing is proportionately lengthened and the fats have of course been subjected not only to prolonged heat, but to intimate contact with the moisture and vapors emanating from the solids associated with them.

Where this variant is employed the resultant cake does not lose sufficient of its gluey properties to press well in a hydraulic press and is better adapted to an expeller.

A patent has been issued along the lines of this method of processing but the system was well-known and used in the manufacture of meat meal and in rendering for years prior to the introduction of the real dry-rendering system.

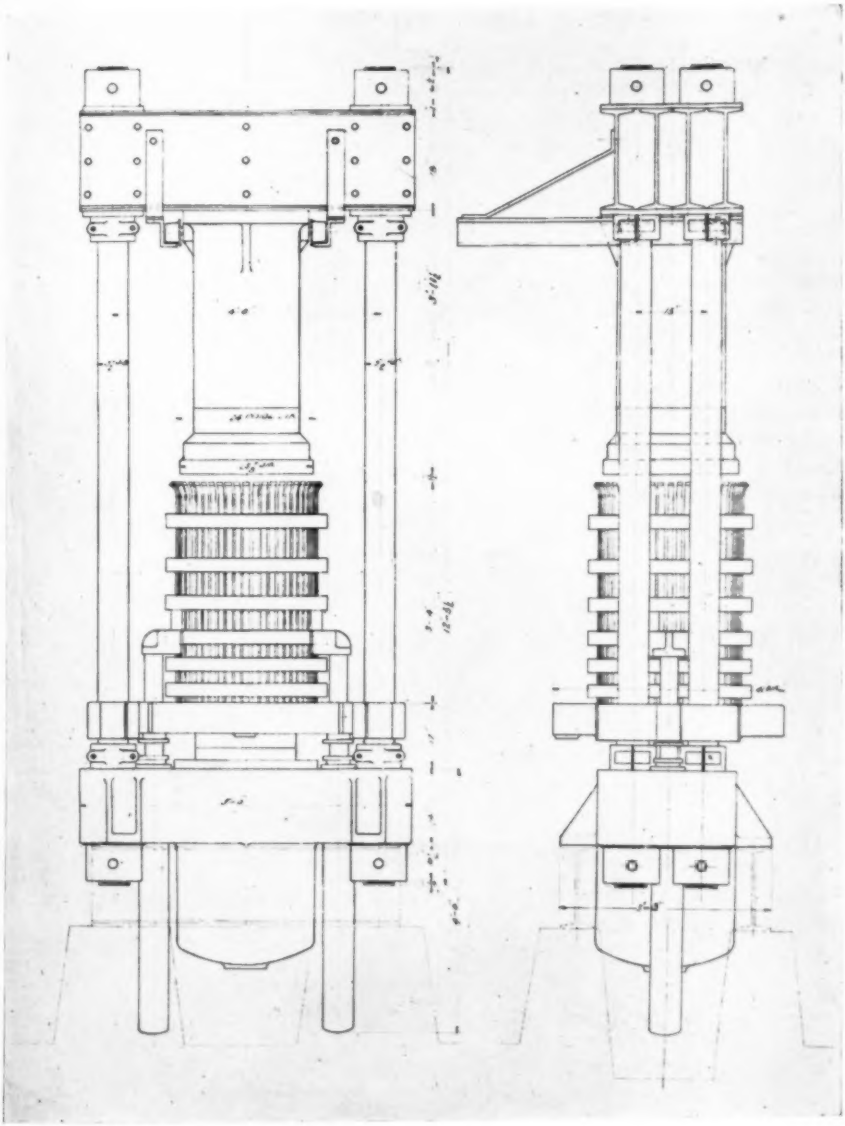
Draining

When cooking has been completed the tallow, lard or grease is drawn off and the solid matter automatically discharged into a false-bottom steel slush box, the free oil draining to the floor or storage-tank and the solid matter or unpressed crackling remaining in the box from which it is loaded into hydraulic press or expeller.

Hydraulic Press

From the standpoint of yield rather than that of quality of tallow or grease the hydraulic press is the most important unit in the dry-rendering system.

On its efficiency depends in large measure the total yield of fats and consequently, a large share of the renderer's profits.



Perfected type of hydraulic press for use in dry-rendering systems

The hydraulic press as used in dry-rendering is of the curb or basket type as illustrated. The drained crackling is loaded into the curb in small batches and while it is still very hot. Only enough material is loaded to make a cake 1″ to 1½″ in thickness when pressed. On top of each such batch a steel spacer plate is placed, another batch added, another plate inserted and so on until the curb is full.

Hydraulic pressure is then applied underneath the ram of the press by means of a steam or power driven hydraulic pump and the entire platen with its load of material is forced upward against the stationary head, which fits inside the curb.

As the pressure increases the fat is forced through narrow slots in the curb and runs down into the lip or trough of the platen and thence by pipe to the storage tank.

Within limits the general principle of pressing is that the greater the pressure the higher the yield. Unfortunately, this principle seems to have been absorbed in the main by the large packers and renderers and not by the small operators.

Good practice would show about 10% fat in ordinary beef cracklings, a result which can be obtained with 500 ton presses of reasonable cost. In general, it would be fair to assume that the smaller renderers will show an average of about 15%, though some will run 30% and above.

The importance of a good hydraulic press can be best appreciated by a study of the chart on next page.

The diagram represents an average 2500 lb. charge of raw material and shows the value of:

(1) The protein content of 312.5 lbs. at $.05 per lb. or $15.63.

(2) The total tallow content of 1187.5 lbs. at $.08 per lb. (no grease in cake) or $95.00.

(3) The various values of the tallow lost in the cakes corresponding to grease analysis of cake.

Ten percent grease in cake is considered good pressing practice. Example:

Suppose a crackling cake analyzes 20% grease. Then our protein value still remains $15.63, but we have lost almost $11.50 worth of tallow (distance from horizontal line to curve) in our cake leaving us only $95.00 minus $11.50 or $83.50 worth of salable tallow.

The curve does not follow the straight line variation because in addition to increased money loss with higher tallow percentages the weight of cracklings also increases materially.

Products from Pressing

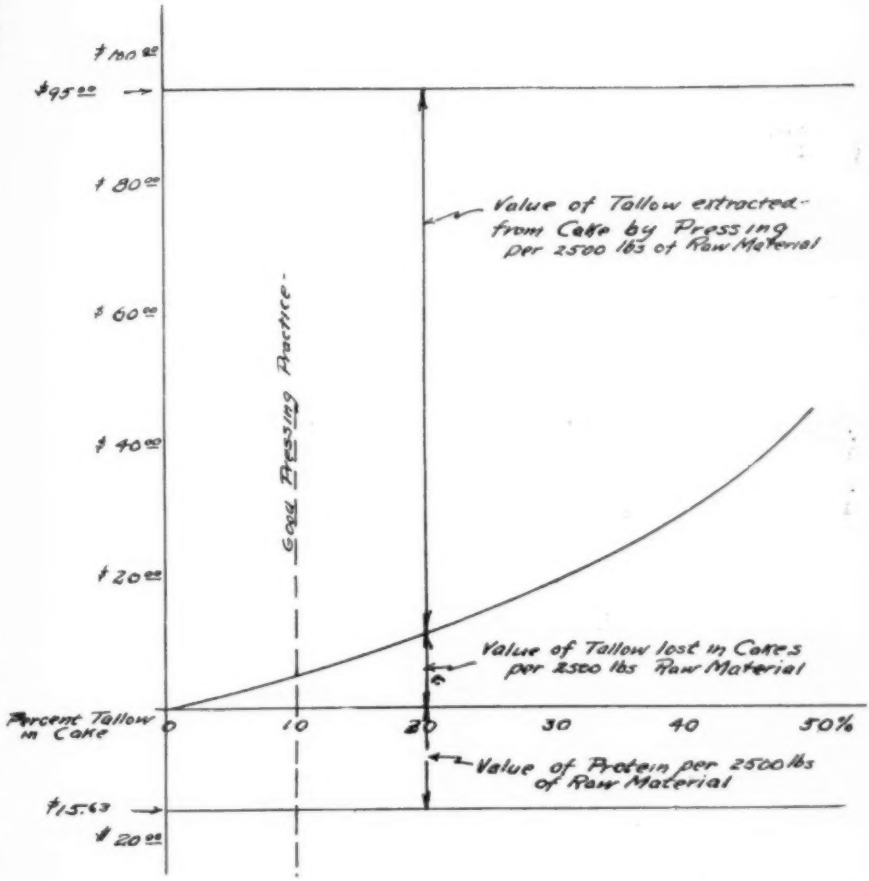
In the dry-rendering system the tallow or grease produced is practically moisture free—consequently, the oils from the melters and the press pass to the same receiver.

After a brief period of settling for precipitation of floating solid particles the tallow or grease is ready for tiercing.

The solid particles which settle to the bottom of the receiving tank are minute pieces of protein material which flow from the melter with the oil or squeeze through the slatted curb of press or expellers.

The volume of these depends on the fineness of raw material grinding; the speed and construction of the melter agitator, the width of opening between the slats of press or expeller curb and the proper cooking of the raw material.

This solid matter is removed from the settling tanks at intervals,



Graphic chart of recoverable values in dry-rendering operation

usually every two or three days, and added to a partially cooked charge of bone or to a charge of fat and bone after the first run of oil has been drawn off. Thus this material reappears in the cracklings and is entirely recovered.

Summary

In general—the dry-rendering process has the following marked advantages over the wet-rendering system, and for these reasons—has come into great popularity in a remarkably short space of time.

1. The dry system is far more rapid.
2. Its products are more valuable.
3. Its products are of higher grade.
4. It requires less labor.
5. It requires less machinery, power and steam.
6. It requires less floor space.
7. It requires less investment.
8. The entire process is tremendously simplified.
9. It is, when properly installed and operated—odorless.
10. It is clean.

Solvent Extraction of the Soya Bean

Discussion of Extraction Methods with Detailed Description of One System

By ANDREW K. SCHWARTZ

SOLVENT extraction of the soya bean has many attractive features, most important of which is the high oil yield obtainable by use of this method of extraction.

Assuming a soya bean of 20% fat and 7% moisture content, the press method gives a cake with 6% fat while the solvent method yields a meal of 0.5% fat content. If cake and meal moisture content were the same at 7% the fat yields per ton of clean bean would be 300 pounds for the press and 392 pounds for the solvent extraction, or 75% and 98% extraction respectively. Based on 11½c oil and $39.00 meal the solvent extraction shows an increased financial return of $8.90 per ton of beans worked. In reality the solvent method shows a greater advantage than indicated because the average soya bean has a lower oil content than 20%—usually around 17%.

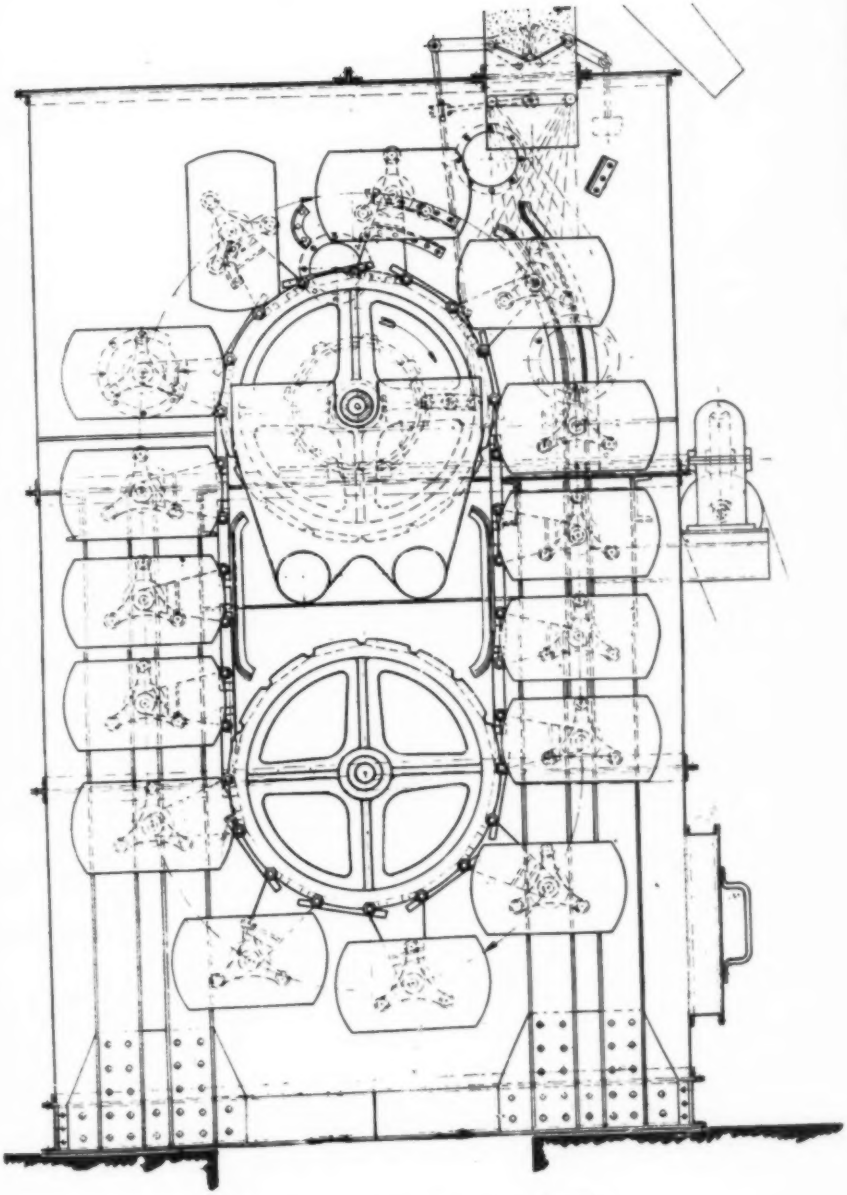
The texture of the soya bean makes it particularly adaptable to solvent extraction. The bean on being crushed gives a somewhat fibrous material, which even with rough handling retains its original crushed shape and size. With proper rolls it may be crushed very fine without the production of any appreciable amount of fines. The crushed material shows no balling or caking action either upon application of slight pressure or when moistened with solvent. Its volumetric shrinkage on extraction is small. In short the soya bean has those properties desired in a material to be extracted by percolation.

The soya bean is unusually rich in vegetable phosphatides and serves as a raw material from which these substances may be recovered.

Particular Method Described

The process described here was patented and developed in Germany, and has been in successful commercial operation there for a period of about ten years. It is based on the principle of continuous operation and counter-current flow and is logically divided into three semi-independent units: Extraction proper, dryer or solvent recovery from extracted meal and oil recovery or distillation unit. One plant is at present extracting soya beans, being operated side by side with Anderson Expellers, the cake of the latter being worked through the extraction process, forming a part of its raw material. Because of this, this particular plant offers a partial comparison of the pressing as against extraction methods.

As shown by the sketch the extractor is essentially a large bucket elevator. The individual buckets, equipped with false bottoms, are filled from a hopper above the extractor, while at the highest point of their flight. They descend on the right side, being simultaneously treated with oil-solvent effluent obtained from the left side of the extractor. On the left and ascending side the material is treated in counter-current with fresh solvent. The bucket, ascending past the



Continuous elevator type extractor

zone of solvent application, goes through a draining period and upon again reaching its highest point is dumped in a hopper below, from where it is delivered to the drying apparatus. The whole is enclosed in an iron casing.

The drying apparatus consists of

a series of specially designed, jacketed tubes, within which the meal is heated and steamed. After traveling the full length of these tubes the meal is delivered through a lock as finished extracted meal.

Evaporators, fractionating columns, heat exchangers, water separator, surface condensers, oil scrubbing tower for solvent recovery from vented air, etc., constitute the recovery unit. The whole is combined as one closed system.

As a solvent a mixture of benzol and alcohol is used. The alcohol enables the recovery of a third and valuable product, "Vegetable Phosphatides."

Operating Requirements and Yields

The following table of six months' operation, representing a run of approximately 8,000 tons, illustrates the quantitative operation of this factory:

No.	Material Worked KG	Loss on Cleaning Beans KG	%	Pressed Oil KG	%	Extracted Oil KG	%
1	1,008,000	7,740	0.8	45,050	4.5	118,960	11.8
2	1,233,500	9,410	0.8	48,190	3.9	150,400	12.2
3	1,330,100	10,360	0.8	47,340	3.6	165,450	12.4
4	1,307,200	11,490	0.9	45,090	3.5	162,170	12.4
5	1,654,900	14,780	0.9	54,580	3.3	211,600	12.8
6	1,380,600	11,330	0.8	42,935	3.1	178,320	12.9
Total	7,914,300	65,110		283,185		986,900	
Average	1,319,050	10,851	0.82	47,197	3.58	164,483	12.38

No.	Phosphatide Oil Emulsion KG	%	Extracted Material Meal KG	%	Finely Ground Extracted Meal KG	%	Total Yield KG	%
1	7,800	0.8	832,575	82.6	200	0.02	1,004,585	99.7
2	8,500	0.7	950,945	77.1	56,550	4.3	1,213,585	98.2
3	9,300	0.7	1,076,520	80.9			1,298,610	97.6
4	14,130	1.1	1,055,725	80.8			1,277,115	97.7
5	18,300	1.1	1,317,800	79.6	10,500	0.6	1,602,280	96.8
6	15,000	1.1	1,076,875	78.1	17,900	1.2	1,331,030	96.4
Total	74,030		6,310,440		85,150		7,727,205	
Average	12,338	0.94	1,051,740	80.0	14,191	1.08	1,287,867	97.5

No.	Required Steam at 8 Atmos. KG	Total Required Power Kw.	Solvent Requirements Benzol KG	%	Alcohol KG	%
1	893,400	53,068	9,500	0.95	7,195	0.71
2	1,067,100	63,107	14,100	1.14	5,150	0.42
3	1,359,500	69,580	14,750	1.11	4,030	0.30
4	1,310,200	62,264	16,100	1.24	6,350	0.49
5	1,317,500	67,294	19,100	1.15	8,620	0.52
6	1,291,400	60,013	14,900	1.08	6,350	0.46
Total	7,239,100	375,526	88,450		37,690	
Average	1,206,515	62,554	14,741	1.12	6,281	0.47

It is apparent from the table that the solvent extraction of the soya bean by this process is an existing commercial enterprise. The table may serve as a basis for both estimating extraction costs and for

comparison with the operation of other existing processes providing certain unique features of this process are taken into account.

As stated above, this process is working side by side with Expellers, the cake of the latter forming part of its raw material. This procedure, aside from the extra power used for pressing, increases power consumption due to extra handling and milling necessitated by it. Then too, it results in supplying the extractor with a raw material of lower oil content. Experience has shown, however, that unpressed beans are as easily extracted as the pressed.

The table is based on the use of a double solvent consisting of alcohol and benzol. The recovery of such a solvent entails a large expenditure of steam, for the actual oil solvent, benzol, is increased by the quantity of alcohol used. The latter, after the first distillation from oil must be separated from the benzol by use of water and subsequently re-distilled and rectified. Of the required steam shown in the table, about 52% is used for the distillation and rectification of the solvents. Using one solvent the steam consumed would be reduced by 50% or the total steam consumption reduced to 75% of the figure given in the table.

The use of alcohol not only adds an extra alcohol loss, but tends to increase the benzol loss, which otherwise would be well under 1%. Actual operation has shown that the use of alcohol is not necessary to complete oil recovery, but that it greatly increases the yield of phosphatides and materially affects the quality of the latter. The phosphatide fraction consists of approximately 15% water, 30% oil and 55% crude phosphatides.

One month's qualitative operation is given in the table below. The averages of daily determinations have been grouped as weekly averages.

Ethyl ether was used as solvent for the fat determinations of cake and meal. With this solvent extracted meal usually shows a 0.5% fat content. Lower figures are often obtained but to consistently extract below 0.5% would unduly increase the extraction period and cut the capacity.

Comparison with Pressed Oils

For the purpose of comparison, representative samples of pressed and extracted oils were carried through the usual refining process with the results given in table on next page.

In comparing pressed with extracted oil it is to be noted that the pressed oil represents the first fraction of expressed oil while the extracted oil is a combination of the oil extracted from the press residue and from the bean. It is seen, however, that the extracted oil is equally as good as the press-

Soya Beans		Pressed Cake		Extracted Meal		Extracted Oil		Pressed Oil	
Fat	Moisture	Fat	Moisture	Fat	Moisture	F.F.A.	Color	F.F.A.	Color
%	%	%	%	%	%	%		%	
17.56	11.00	12.95	10.07	0.86	14.50	0.29	6.2	0.15	5.8
17.47	12.10	12.50	11.23	0.48	15.30	0.28	6.2	0.11	5.8
17.47	11.10	12.70	11.20	0.78	14.60				
16.90	10.80	12.10	10.00	0.53	13.00				
17.30	11.40	11.80	10.60	0.50	13.60				
Monthly average									
17.30	11.30	12.70	10.60	0.67	13.90	0.29	6.2	0.14	5.8

Comparative Analyses and Refining Results on Pressed and Extracted Soya Bean Oils

	Extracted Oil	Pressed Oil
Percentage volatile matter	0.43	
F. F. A.	0.73	0.30
Color (Lovibond)	35y, 16.5r, 2.5b	35y, 9.8r
Iodine No.	134.7	133.8
Loss of refining	3.6%	3.3%
Color	35y, 9.6r	35y, 7.2r
6% Pikes Peak Bleach	28y, 2.8r	22y, 2.2r
Deodorized color	7y, 0.7r	7y, 0.7r
Flavor	Good	Good
Cold test	Better than 48 hrs.	Better than 48 hrs.

Another sample compared as follows:

	Extracted Oil	Pressed Oil
Percentage volatile matter	0.13	0.08
Iodine No.	122.8	122.4
F. F. A.	0.70	0.35
Sp. gr. 15° C.	0.9262	0.9293

Still another sample compared as follows:

	Extracted Oil	Pressed Oil
Color	35y, 11.3r	35y, 10.6r
F. F. A.	0.70	0.35
Iodine No.	132.6	132.2
Moisture per cent	0.13	0.08
Total fatty acids	94.59	94.73

ed for both technical and edible purposes.

Typical analyses of extracted meals are as follows:

Moisture	Fat	Protein	Fibre	Ash
8.60	0.90	47.40	3.53	
11.90	0.80	48.30	5.00	5.10
13.50	0.40	47.20	4.70	5.40
15.53	0.48	48.45	5.00	5.40

Extracted meal is solvent free. It is from white to light straw in color and when finally milled has much the same appearance as wheat flour. Aside from its use as a cattle feed, for which it is highly valued, it is regularly used in Germany for human consumption, as such, and incorporated with such materials as cake chocolate, soup cubes, wheat flour for bread making, etc. In Europe this meal commands a premium over pressed soya meals, although this is contrary to American preference.

OIL & FAT INDUSTRIES at the Chemical Exposition

ALL readers and other friends of OIL & FAT INDUSTRIES who intend to visit the Eleventh Exposition of Chemical Industries, which will be held at the Grand Central Palace, New York, Sept. 26 to Oct. 1, inclusive, are invited to make their headquarters at our booth, Number 525, or at our office at 220 West 42d St. The Chemical Industries Exposition this year will be bigger and better than ever and we hope all our readers will attend. Don't forget the number of our booth, 525.

Liquid Caustic Soda

A Discussion of Advantages of Handling This Essential Chemical in Liquid Form

By ROBERT J. QUINN

DURING the past half-dozen years a change of economic importance has been taking place in the method of distributing caustic soda to large industrial consumers. Many thousands of tons formerly shipped in solid form in drums are now being delivered to the users in tank cars in the form of a solution containing about equal parts of water and caustic soda. Although the tonnage shipped in this liquid form is increasing rapidly every year, it still represents only a part of the total consumption of those users of caustic soda who can be served economically in this manner.

Robert J. Quinn

The purpose of this article is two-fold:—first, to present to those interested in a possible change from solid to liquid caustic, facts which will enable them to judge the benefits of such a change in their own plants; and second, to place in the hands of those now using liquid caustic, some useful data on approved methods for its transfer, handling and storage, together with some notes on the dilution, testing and properties of caustic liquors. This material has been selected for its practical value to the plant executive and operating staff and is based upon the wide experience of chemists and engineers and upon information from authoritative reference sources.

Why Liquid Caustic Soda?

A brief review of the operations in the manufacture and handling of caustic soda, both in solid and in liquid form, will make it clear that the increasing use of liquid caustic is a sound economic development. In the manufacturing process caustic soda is first obtained in the form of a water solution containing from 10% to 25% of sodium hydroxide. To produce solid caustic, it is necessary to drive off *all* the water in this solution by evaporation processes which consume a large amount of fuel. Drums must then be provided for the fused caustic and labor expended in filling, handling and loading these containers for shipment. At the consumer's plant these operations are reversed, the solid caustic being made once more into a water solution with the attendant cost of

unloading, handling and cutting open many drums, dissolving the caustic and disposing of the worthless empty drums.

In the case of liquid caustic soda the evaporation process is carried only to the point where the original dilute solution has been "boiled down" to a concentration of approximately 50% by weight of caustic soda. In this form it is shipped to the consumer in tank cars and handled mechanically by means of suitable piping connected to the car. In the consumer's plant the 50% caustic liquor is readily diluted to the desired concentration.

In furnishing liquid caustic the manufacturer's processing and handling costs are reduced to a minimum, permitting him to offer caustic in this form at a lower price. The user thus has the advantage of a substantial reduction in price over caustic soda in solid form, as well as the economy and convenience of handling his caustic entirely by mechanical means.

Who Can Use Liquid Caustic?

Whether or not a plant may profitably use liquid caustic soda is determined by the following factors: —(1) delivered cost on solid and on liquid, (2) yearly consumption, and (3) cost of handling solid caustic.

In discussing delivered costs, there are several facts to be kept in mind regarding solid and liquid caustic soda. First, as to works prices, there is usually a price differential of 35 cents per 100 pounds in favor of caustic soda in liquid form. Second, the freight rate between two given points on solid caustic does not always apply on liquid caustic as well; in some territories both rates are the same and in others a lower rate applies on liquid caustic, with a varying differential between the two rates at different points. Third, in transporting every 100 pounds of caustic soda in the form of 50% liquor, 100 pounds of water are also carried. In other words, for every 100 pounds of caustic soda received in the form of 50% solution, the consumer pays freight charges on 200 pounds of material.

Some examples of delivered costs, will serve to make this clear:

CASE A—Rate on solid caustic 19c; on liquid 19c

	Solid	Liquid
Price per 100 lbs. f.o.b. works	$3.00	$2.65
Freight per 100 lbs. caustic soda	.19	.19
Freight per 100 lbs. water	.00	.19
Delivered cost per 100 lbs.	$3.19	$3.03

Differential in favor of liquid caustic—16c per 100 lbs.

CASE B—Rate on solid caustic 38½c; on liquid 25c

	Solid	Liquid
Price per 100 lbs. f.o.b. works	$3.00	$2.65
Freight per 100 lbs. caustic soda	.385	.25
Freight per 100 lbs. water	.00	.25
Delivered cost per 100 lbs	$3.385	$3.15

Differential in favor of liquid caustic—23½c per 100 lbs.

CASE C—Rate on solid caustic 45½c; on liquid 45½c

	Solid	Liquid
Price per 100 lbs. f.o.b. works	$3.00	$2.65
Freight per 100 lbs. caustic soda	.455	.455
Freight per 100 lbs. water	.00	.455
Delivered cost per 100 lbs.	$3.455	$3.560

Differential *against* liquid caustic —10½c per 100 lbs.

It will be noted from these examples that the freight rates on both solid and liquid caustic must be known in order to work out a comparison of the delivered costs in these two forms.

An item in the delivered cost of the solid material, not included in the preceding examples, is that of freight on the tare weight of solid caustic drums. This amount is small, but should not be overlooked. It will vary according to the solid freight rate from a little more than half a cent per 100 pounds in Case A to one and one-third cents per 100 pounds in Case C.

In general, provided the delivered cost is favorable, a consumer purchasing solid caustic, in carload lots, may profitably take deliveries of 50% caustic liquor, in tank cars. An 8,000-gallon tank car of 50% caustic liquor contains approximately 25 tons of caustic soda in solution, or the equivalent of a minimum carload of solid caustic in standard 700-lb. drums. On this basis a buyer of several carloads of solid caustic per year may be considered a potential user of 50% liquid caustic.

It will be obvious that the consumer of liquid caustic soda must have access to railroad siding facilities for the unloading of tank cars. A siding at some distance from the plant may be conveniently used, however, since the liquor is transferred from the car into storage by means of piping. The cost of installing transfer equipment may be offset in a short time by the economy of mechanical handling in place of trucking and manual handling of solid caustic.

Handling solid caustic soda, including unloading and breaking open the containers, dissolving the caustic and disposing of the worthless empty drums, costs approximately 12 cents per 100 pounds in the average plant. This figure is based on conservative estimates and will vary only in accordance with handling methods and local wage-scales on labor. Handling is a very small item with caustic liquor; the change from solid caustic will therefore effect a saving in handling cost of around 10 cents per 100 pounds. This is in addition to any saving that may be obtained in delivered cost.

Even where no money saving can be shown for liquid caustic through a consideration of delivered costs and handling costs, the greater convenience of using caustic soda in liquid form makes it attractive to large consumers. The great increase in shipments of liquid caustic soda within the past few years indicates that growing numbers of progressive consumers are recognizing the economy and convenience of this commodity. Consumers served in this manner represent all the principal industries using caustic soda—soap makers, petroleum and vegetable oil refiners, paper mills, manufactures of chemicals, rayon and liquid bleach, textile bleachers and mercerizers.

Purity of Liquid Caustic

In addition to the advantages it frequently offers from a cost standpoint, caustic soda in liquid form is ordinarily a purer product than the solid material in drums.

At Niagara Falls one large electrolytic plant produces an extremely pure liquid caustic which is known as "cell liquor" because obtained directly from the electrolytic cells without evaporation or processing of any kind. This so-called cell liquor is discharged from the cells at a concentration of from 25% to 30% by weight of caustic

soda. Although this solution has a limited radius of distribution due to the greater amount of water that must be transported, it offers a highly desirable form in which to handle the commodity on account of the very high purity of the caustic it contains and because of the low freezing-point of the liquor. It is an interesting fact also that this product is the only caustic liquor that can be shipped as originally produced without subsequent processing. Liquors from some types of electrolytic cells are so low in concentration of caustic and usually contain such large amounts of salt in solution that they cannot be shipped as originally produced. The original liquor obtained from the ammonia-soda process is also too low in concentration of caustic to permit its economical distribution.

The following are typical analyses of the several concentrations of liquid caustic soda as shipped from the two different plants of one producing company, one of the country's largest shippers of caustic.

Percentage Content of:	Electrolytic Cell Liquor	Caustic Liquor Concentrated	Soda Process Caustic Liquor
Sodium Hydroxide	27.5	49.8	50.07
Sodium Carbonate	0.10	0.25	0.35
Sodium Chloride	0.05	0.10	0.29
Sodium Sulphate	0.008	0.016	0.21
Sodium Sulphide	(None)	(None)	(None)
Silica	0.005	0.007	0.14
Iron Oxide & Alumina	0.002	0.008	0.034
Calcium Oxide	0.003	0.005	0.005
Magnesium Oxide	0.001	0.002	0.007

The figures in the first column represent an average analysis of cell liquor. In the second column is shown an analysis of the 50% caustic solution obtained through partial evaporation of this cell liquor. It is of interest to note the low salt content of these liquors. In both the cell liquor and the concentrated solution, the sodium hydroxide content of the caustic soda is in excess of 99%. The figures in the third column represent an average analysis of the liquid caustic produced by the ammonia-soda process.

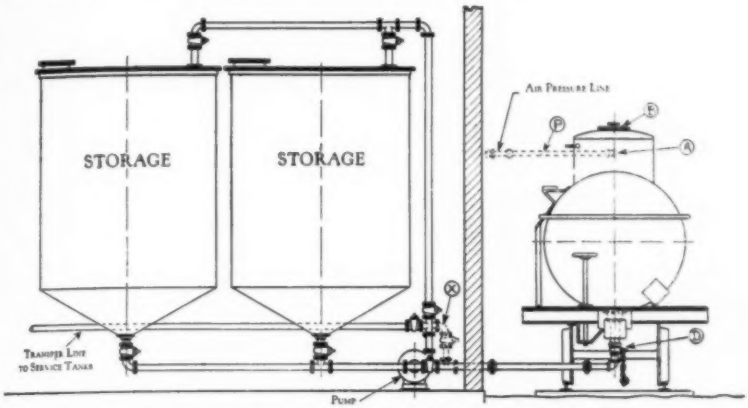
Tank Car Equipment

Liquid Caustic Soda is shipped in tank cars of 8,000 gallons capacity (equivalent to approximately 100,000 pounds of 50% caustic liquor). This equipment is of the latest type, conforming to A.R.A. Specifications for Class III Tank Car and to I.C.C. Specification No. 103.

All tank cars for caustic liquor should be equipped with steam heating coils, for use during the winter months, and with an auxiliary bottom outlet valve. Steam heating coils should be of 2-in. extra-heavy steel pipe, with 2-in. connections, and all joints welded throughout to prevent leakage sometimes experienced with threaded joints.

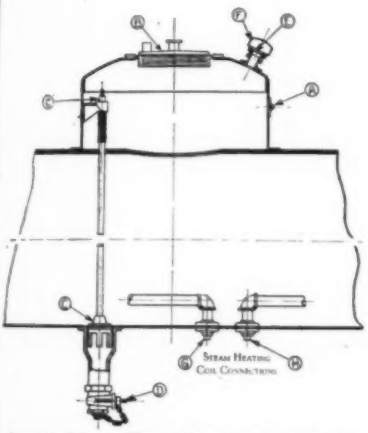
The auxiliary bottom outlet valve mentioned above was adopted several years ago by a large shipper as a protective measure for users of caustic liquor. It had been found next to impossible to maintain the regulation Tank Car Bottom Outlet Valves tight against leakage be-

Approved Methods for Unloading Liquid Caustic Soda



Typical Layout for Unloading Liquid Caustic by Pump and by Air Pressure

NOTE: Additional Equipment for Unloading by Air Pressure Indicated by Dotted Lines, Marked "P" and "X"



Longitudinal Section Through Dome and Bottom of Car

Instructions for Unloading

By Pump.

1–Attach two-inch pipe at valve (D) by means of flanged connections.
2–Remove plug (A) for release of internal pressure.
3–Remove dome cover (B).
4–Open valve (C) (Inside dome).
5–Open valve (D) and start transfer pump.

By Compressed Air.

1–Attach two-inch pipe at valve (D) by means of flanged connections.
2–Remove plug (A) and connect compressed air line (P) at this point.
3–Remove dome cover (B).
4–Open valve (C) (Inside dome).
5–Replace dome cover (B) (Tight gasket).
6–Apply air pressure (not to exceed 30 lbs. per sq. inch).
NOTE: Lead disc (E) in vent (F) ruptures at 30 lbs. per sq. inch air pressure. This is sufficient to lift 50% caustic liquor 35 ft. above R.R. track level.
7–Open valve (D) and other valves in line to storage.
NOTE: When unloading by means of compressed air, by-pass connection (X) with valve must be provided.

cause of their location and the requirements imposed by I.C.C. specifications. Such leakage resulted in the discharge nipple becoming filled with caustic liquor, which very often spilled over the workmen when the cap was removed. To prevent this, a change in the I.C.C. specifications was obtained permitting the installation of the auxiliary bottom discharge valves on tank cars for caustic liquor service. All caustic liquor cars are inspected on the completion of each trip, washed out, tested, and the valves ground when necessary. During the cooler months the steam heating coils are tested on the return from each trip.

This careful maintenance of tank

car equipment insures the customer against transportation delays and against contamination or loss of material.

Transfer Equipment

Liquid caustic is transferred to storage tanks by means of a pipe line connected to the car at a point on the consumer's railroad siding. The two approved methods for unloading into storage are by means of pump and compressed air. These methods are shown in connection with the sectional drawings on the previous page. In rare cases where storage tanks are located below the siding track level, caustic liquor may be transferred by gravity, with the same procedure as for unloading by pump. It is also possible to unload by suction, through the manhole opening, but this practice is not recommended.

Lubricated plug cocks or valves, transfer pumps and pipe lines should be of all-iron construction.

Storage Equipment Specifications

CAPACITY:

To conform with consumer's requirements; minimum capacity approximately 16,000 gallons.

PLATES:

Of flange steel having ultimate strength of 55,000 lbs. per sq. in.

RIVETS:

Of boiler rivet steel driven hot.

JOINTS:

To be double riveted and calked inside and outside.

Winter Handling and Storage

Concentrated solutions of caustic soda (25% to 50% NaOH content) will deposit crystals of the solid hydrates of sodium hydroxide under normal winter weather conditions. At abnormally low temperatures, around 0° F., and depending upon the concentration of the solutions, complete solidification may take place.

This consideration necessitates the use of steam coils in tank cars and in storage tanks exposed to winter weather. Outside pumps, transfer lines, etc., must be effectively protected from the cold.

During the winter months the physical condition of liquid caustic should be noted before transfer from car or storage is attempted. An examination can readily be made by probing the liquid with an iron rod for indications of solid hydrates or to determine the extent to which solidification has progressed. If a top crust has formed, it must be broken before steam is applied in the heating coils in order to provide for expansion of the heated liquid. The steam pressure applied should not exceed 10 lbs.

Where storage is provided within heated buildings steam coils inside the tanks are not required. Caustic liquor of 50% concentration is more conveniently handled in this type of storage. When 50% liquor is stored in outside tanks, it is necessary to maintain heat in the coils almost continuously during the winter months. Less difficulty is experienced with outside storage in the case of more dilute solutions of 25% concentration or less. Shipments usually consist of 50% liquor which requires a dilution with 1.4 volumes of water to reduce the concentration to 25% sodium hydroxide. A tank of 20,000 gallons capacity would be required for storing the contents of one tank car (8,000 gallons) of 50% liquor after such a dilution.

Heat of Dilution

When water is added to liquid caustic soda, heat is evolved and a rise in the temperature of the solution takes place. This is due to

the heat of reaction in the formation of the various hydrates of sodium hydroxide, and is generally termed heat of dilution. This heat effect is considerable and allowance should be made for it in diluting caustic liquor, particularly if the diluted solution is to be used in the preparation of bleach liquor.

In general, a sharp rise of temperature will be noted until a volume of water has been added about equal to the original volume of the liquor. From this point on, further dilution results only in a decrease in temperature. The extent of these temperature changes is indicated by the results of the following dilutions in a Dewar flask.

One volume of a solution containing 50% NaOH by weight was diluted with successive portions of distilled water each equal to one-half the original volume of 50% solution. The initial temperature of both solution and water was 14.5°C. (58.1°F.), and the temperature of the solution was observed following each dilution. After dilution (1) the temperature was 38.5°C. (101.3°F); (2) 45.5°C. (113.9°F.); (3) 46.5°C. (115.7°F.); (4) 44.8°C. (112.6°F.); (5) 42.5°C. (108.5°F.); (6) 40.0°C. (104.0°F.).

Data based upon laboratory experiments is only of value, however, in estimating the temperature increase that may be expected upon diluting caustic liquor in the plant. The size and shape of dilution tanks, their heat capacities and their heat radiations, all exert such an important influence that theoretical figures cannot be applied in actual practice. It is advisable for the operating man to determine this factor by observation under plant conditions.

Sampling of Caustic Liquor

Careful sampling is absolutely essential to the accurate analysis of tank car shipments of liquid caustic. Particularly in cold weather, it is important that heating be continued until all crystals have gone into solution and the entire contents of the car has been converted into a liquid condition. Samples containing crystals of hydrates of sodium hydroxide will obviously not be representative of the liquor in the tank car. Since some of these hydrates may form in concentrated liquors even under mild weather conditions, it is desirable that 50% liquid caustic be warmed until a surface temperature of approximately 75° F. has been reached.

Samples should be taken at three different depths in cars of cell liquor (27.5% caustic soda) and at five different depths in more concentrated liquors. The best method of sampling is by means of an iron or wooden rod, fitted at one end with a basket in which is fastened a small-necked 16 oz. bottle. This is provided with a cork attached to a wire, so that it can be removed when the bottle has been submerged to the proper level. Separate samples are thus taken at various depths in the car.

Analysis of Caustic Liquor

TOTAL ALKALI:

If no determination for carbonate is to be made, weigh out directly in a closed weighing bottle about 1.5 grams from the sample of 50% liquor, or a correspondingly larger amount for liquors of less concentration.

The weighed sample is transferred to a beaker and titrated with N/2HCl in the presence of methyl orange. This titration (A) is a

measure of the total alkali present and may be expressed in terms of Na_2O and NaOH.

$$\frac{A \times 3.1}{\text{Wt. of sample} \times 2} =$$

% Total Alkali as Na_2O

$$\frac{A \times 4.0}{\text{Wt. of sample} \times 2} =$$

% Total Alkali as NaOH

SODIUM HYDROXIDE AND SODIUM CARBONATE:

When a determination for carbonate is to be made, a larger sample of from 14 to 16 grams of the 50% liquor is used. The weighed sample for analysis is diluted to the mark in a 500 cc. volumetric flask with water free from CO_2. Air should be excluded as much as possible during this operation.

Transfer a 50 cc. aliquot of the sample and add 10 cc. of a 10% solution of $BaCl_2$. Using one drop of phenolphthalein for an indicator, titrate with N/2HCl to almost an end point. After washing down flask with distilled water (free of CO_2), an additional drop of the indicator solution is added and the titration then continued to the end point. For extreme accuracy, air free of CO_2 should be introduced into the flask during titration. This titration (B) is a measure of the alkalinity due to sodium hydroxide.

$$\frac{B \times 4.0}{\text{Wt. of sample in aliquot} \times 2} =$$

% Actual NaOH

The difference between the two titrations (A) and (B) is a measure of the Na_2CO_3 present.

$$\frac{(A - B) \times 4.3}{\text{Wt. of sample in aliquot} \times 2} =$$

% Na_2CO_3

The figure for total alkali as Na_2O, calculated from the preceding method of analysis is the true or actual sodium oxide (Na_2O) content. To obtain the percentage of total alkali as Na_2O according to the New York and Liverpool test, multiply the figure for total alkali as Na_2O, as determined from analysis, by the factor 1.0323.

Selling Basis on Liquid Caustic

During the first few years after caustic soda in liquid form was placed on the market, there were many different practices in use as regards specifications and prices. Much confusion existed in the minds of consumers, who found it difficult to compare the product and price offered by one producer with that offered by another. The situation was further complicated by the fact that liquid caustic was demanded by consumers in a variety of concentrations ranging from 25% to 50%, none of which was a standard article of commerce.

Through the co-operation of progressive manufacturers and consumers, liquor of 50% concentration has now been established as the standard for the commodity liquid caustic soda.

It was also desirable that some standard practice be adopted in marketing and invoicing liquid caustic, and that this practice be based as far as possible on terms familiar to the buyer. Most caustic liquor is now sold on the basis of its caustic soda content expressed in terms of solid caustic soda of standard test, that is, 76% actual Na_2O. Contracts usually specify a given tonnage of "solid caus-

tic soda (76% actual Na_2O) to be shipped in the form of 50% solution." Buyers can now compare on a common basis their quotations from various producers, or check their price on caustic in liquid form against past market levels on solid caustic.

The following rule may be used for computing the value of a car of liquid caustic:

$$\text{Wt. of liquor} \times \frac{\%\ \text{Total Alkali as NaOH}}{\%\ \text{Purity of Caustic}} \times \text{Price} = \text{Value of Car}$$

For example: A shipment consisting of 100,000 lbs. of liquor is found by analysis to contain 49.00% NaOH. The market price on solid caustic (76% actual Na_2O) is $3.00 per 100 lbs.; liquid caustic is 35 cents lower in price, or $2.65 per 100 lbs.

$$100{,}000 \times \frac{49.00}{98.00} \times \$0.0265 = \$1{,}325.00$$

The average 76% solid caustic soda contains approximately 98% NaOH and 2% of solids other than NaOH. In the above rule, the term "% Purity of Caustic" refers to this average figure 98%. In special cases where the purity of the caustic furnished averages above or below this figure, the actual percentage purity of such caustic should be inserted.

President Trevithick Names Additional Committees

Neutral Oil and Detergent Bodies to be Headed by McLean and Howells Respectively

PRESIDENT TREVITHICK of the American Oil Chemists' Society, in a communication to the Editor of OIL & FAT INDUSTRIES, announces the personnel of the following committees of the Society for the year 1927-1928:

Refining Sub-committee A
(Neutral Oil Committee)
Claude E. McLean, Chairman,
c/o Choctaw Cotton Oil Co.
Ada, Oklahoma.
George S. Jamieson
M. G. Boulware
E. C. Ainslie
A. S. Richardson
F. R. Robertson

Detergents Committee
F. H. Guernsey, Chairman,
c/o Cowles Detergent Co.
Lockport, N. Y.
L. T. Howells, Vice-chairman
H. C. Bennett
C. P. Long
W. T. Reese
J. S. Boulden
E. T. Marceau
A. S. Richardson
V. K. Cassady
E. R. Millard
F. H. Rhodes
A. K. Church
H. S. Mitchell
F. W. Smither
T. G. Vail
L. F. Hoyt
W. C. Preston

Due to the fact that Mr. Hoyt will be exceptionally busy this year, he found it necessary to decline re-appointment as chairman of the Detergents Committee.

What Is a Refined Oil

Various Definitions and Classifications Evaluated and Compared

By HERMAN ASPEGREN

THE subject "What is a Refined Oil" is of rather large scope, because refined oil includes all kinds of oils used for edible as well as for technical purposes. My knowledge and experience has been principally with cottonseed oil, and, therefore, I will confine my remarks to this specific oil.

To refine means to "purify." The meaning of purification varies greatly, however, depending upon not only for what purpose the oil is used but also in what country the refining is being done.

A "refined vegetable oil" may mean an oil simply purified by the method of settling, or it may mean an oil treated with acid, or it may mean an oil purified with an alkali, filtered or bleached with fuller's earth, or a similar filtering medium. It may also mean an oil which has not only been neutralized and filtered but deodorized; it may mean a winterized oil or a hydrogenated oil. Someone may insist that an oil is not properly refined if he does not agree with the flavor and odor of same. Others may say that an oil is not properly refined if it contains more than a certain amount of free fatty acid, and in this way there are many ideas of what constitutes a properly refined cottonseed oil.

Traders' Definition

On the Exchanges of this country where refined cottonseed oils are traded in and among vegetable oil chemists and traders I believe by "refined oil" is generally meant an oil which has been alkali refined and filtered.

The advancement of science has made it possible to further treat, or I might say further refine or process cottonseed oil and thereby produce oils more suitable for many purposes than the oil which has only been alkali refined. Not only color, taste or odor, but even the chemical and physical characteristics of such oils may differ to quite some extent from the characteristics of the oil which has undergone no other treatment than alkali refining, in view of which some large consumers when making their purchases of refined oils sometimes demand specifically some tests with which the refined oil must conform.

To buy according to such rigid specifications is not, however, the rule but rather the exception, most of the refined cottonseed oil today being bought either according to the grades as defined by the Interstate Cottonseed Crushers' Association, New York Produce Exchange, or New Orleans Cotton Exchange; or according to the trade names given to the refined oil by the various manufacturers.

The refining of a crude oil is done in many steps. The first process is elimination of substances held in suspension or emulsion, coagulating the albuminous matters, saponifying the fatty acid and resinous matters, destroying enzymes, removing to a certain extent the coloring matters, etc.

The first step in the refining process can be carried out in several ways, however, in this country still today is probably universally done by means of adding caustic soda to the crude oil under agitation and heating, later decanting the refined oil, settling, and finally filtering it with a filter medium, usually fuller's earth.

The refined cottonseed oil thus produced was years ago used not only for technical purposes, but very largely also for edible purposes. Today, however, comparatively little cottonseed oil of this quality (that is not further treated) is used for edible purposes.

However, the trade rules as adopted by the New York Produce Exchange, Interstate Cottonseed Crushers' Association and New Orleans Cotton Exchange evidently still refer principally to cottonseed oil not further treated, and in fact the New Orleans Cotton Exchange specifically prohibits the tendering of deodorized refined cottonseed oil.

Lack of Uniformity in Rules

The Interstate Cottonseed Crushers' Association and New York Produce Exchange clearly specifiy that caustic soda shall be used for making laboratory analyses of crude cottonseed oil, but nothing is mentioned about how a refined oil should be produced on a commercial scale, and it is evidently left to the individual refiner to apply whatever refining method he desires.

The rules do not specifiy what makes an oil a "refined" oil, but in defining the various grades of refined oil, however, certain limitations as to color and percentage of free fatty acid are mentioned; further, it is in most cases positively stated that the oil shall be free from water and settlings.

I have tabulated the specifications for the various grades of refined cottonseed oil according to the trading rules of the Interstate Cottonseed Crushers' Association, New York Produce Exchange and New Orleans Cotton Exchange, and it is apparent from the table that some of the grades mean different things, according to under what rule the grading is done; for instance, the grade "Prime Summer Yellow" means different qualities according to the Interstate, Produce Exchange or New Orleans Cotton Exchange rules, and in the same way the grade "Bleachable Prime Summer Yellow" does not necessarily mean the same according to Interstate's rules and New Orleans Cotton Exchange Rules as compared with Produce Exchange rules.

The grade "Good Off Summer Yellow" means the same according to Interstate's rules and Produce Exchange rules, but does not necessarily mean "Good Off Summer Yellow" according to New Orleans Cotton Exchange rules.

The difference in the meaning of the same grade is visible from the tabulated statement on page 302.

Interstate Cottonseed Crushers' Association and New York Produce Exchange rules further specify that the refined oil produced from extracted crude oil is not a good tender unless the nature of the oil is declared at the time of sale.

We are often inclined to consider the expression "Refined Oil" synonymous with a "neutral oil" so far as free fatty acid is concerned. The trade rules, as you know, however, tolerate an appreciable amount of free fatty acid even as high as three-quarters of one percent in reddish off oil.

Therefore, a refined oil may legitimately contain even a higher percentage of free fatty acid than is found in good crude oil.

Water in the refined oil, as I have already mentioned, is not permitted, and I suppose by water is meant such water which can be detected in the form of cloudiness or drops with the naked eye, as mostly all commercially refined oils tendered on Exchange contracts contain moisture, sometimes more and sometimes less, but probably always some. Unfortunately, the official moisture test is very unsatisfactory. In rule 272 of Interstate Cottonseed Crushers' Association's rules there is given a description of how to make a test on moisture or volatile matter. The title of the rule indicates that the loss in weight may be due to not only moisture, but also to volatile matters. In spite of same the instructions are to report the loss as moisture.

Evidently what the Chemist has to report as moisture in many cases is no moisture at all. For that reason I believe that many users of refined cotton seed oil misunderstand or give too great importance to this so-called moisture test.

In the beginning of my remarks I mentioned that when different consumers talk about a properly refined oil they may have in mind entirely different qualities of oil. For instance, those still today who use refined vegetable oil as lamp oil look at the refining process not from the point of view of neutralizing the free fatty acids, but principally from the point of view of removing the impurities in suspension or emulsion, and they are especially interested in having a refined oil free from soap in solution.

Various Steps of Refining

The alkali refining process does only to a limited extent affect the constituents of the oil which are responsible for its specific flavor or odor, but the consumers of refined oil for edible purposes desire oils free from all flavors or odors if possible, and are only in special cases willing to use alkali refined oil not further treated. Even if the oil is sweet in flavor and odor, which is the expression used in the rules for prime oil, still the peculiar taste or odor which is characteristic of cottonseed oil is present and is often objectionable. Through the deodorization process, which as you know, consists of blowing super-heated steam under vacuum through the hot oil the flavor or odor of the refined oil is partially or entirely eliminated. The deodorization process can be carried out more or less successfully, and therefore when a consumer of edible oil talks about "properly" refined or "neutral" oil, he has principally in view a well-deodorized oil which either has no taste or odor at all, or if any, a pleasant one. The deodorization process does not change materially, as a rule, the principal characteristics of a cottonseed oil, nor the chemical composition of same, but nevertheless the effect is far reaching and the deodorization process I believe should be considered as part of the refining process.

Very large quantities of refined cottonseed oil are being used in the manufacture of shortening. Not only is it important that such an oil be well deodorized, but it is very important that the oil be of a light color. And "properly" refined oil for this trade therefore in addition to alkali refining and the deodorization process must as a rule be treated with a bleaching

medium such as raw or acid treated fuller's earth, or activated carbon. The effect of the bleaching medium on the refined oil has not yet been sufficiently studied. Probably some of its effects are due to the physical absorption of colors and some effect is due to chemical reaction. No doubt the bleaching should be considered part of the refining process.

When cottonseed oil is to be used by the margarin trade, an excellent flavor and odor of lasting quality is paramount, but the color of the oil also plays an important role. Some margarin manufacturers desire a very light oil, when again others desire a dark colored oil. All depending upon what color of the oil is desired, a properly refined cottonseed oil for the margarin trade may mean a well deodorized, either light or dark colored oil.

Phosphatides Lower Quality

Most all cottonseed oils which have been alkali refined, filtered and deodorized, but not further treated will develop a deposit of a light color if left standing in a bottle, especially when exposed to the light. I do not mean a deposit caused through the precipitation of the solid glycerides, but I refer to a deposit which it appears is caused by the presence of phosphatides, and we often hear complaints that an oil showing such a deposit is not properly refined. Of course, we know that in percentage such a deposit is entirely negligible and that the deposit is entirely due to the chemical nature of the oil. However, in spite of this some consumers of oil do not consider a cottonseed oil properly refined if upon standing it develops such a deposit, and the elimination of the phosphatides may sometimes become part of the refining process.

In the winterizing process some of the solid glycerides, and probably the phosphatides are separated and removed from the refined oil through a chilling and filtering process. Depending upon the length of time winterized oil will stand clear, brilliant and limpid when exposed to the so-called Winter Oil Test, an oil is more or less suitable for the trade desiring this grade of oil, salad oil and mayonnaise manufacturers, and in spite of the fact that the trade rules prescribe that such an oil does not necessarily need to stand more than five-and-a-half hours clear and brilliant, some consumers desire an oil which will stand a considerably longer time clear and brilliant, and unless it stands the test considerably longer do not consider a cottonseed oil properly refined for their purpose. No doubt the winterizing process should be considered a part of the refining process in spite of the fact that some of the characteristics of the oil, such as iodine number and titer, are materially changed in the process.

In the mind of a dough or cruller baker when he talks about a properly refined cottonseed oil is principally the smoke point. A refined oil, well deodorized with high smoke point and which does not readily give off acrolein vapors is desired. The color of the oil, however, does not seem to be of real importance.

Effects of Hydrogenation

In the hydrogenation process some of the unsaturated glycerides are converted into less unsaturated or entirely saturated glycerides, with the result that different qualities of the original oil are developed. For the cracker baker a hy-

Comparison of Cottonseed Oil Specifications of the Three Trading Exchanges

GRADING		Free From		Color		Bleaching	Max.	
Interstate C. S. C. Assn.	N. Y. Produce Exchange	New Orleans Cotton Exchange	Water and Settlings	Flavor and Odor	Not Darker Than	Not Lighter Than	Not Darker Than	Free Fatty Acid	Cold Test
C.S.Y.			" cl b	Sweet	35Y–7.6R			⅛	
P.S.Y.			" cl	Sweet	35Y–7.6R			¼	
	P.S.Y.		" cl	Sweet	35Y–7.6R	35Y–3.5R		¼	
		P.S.Y.	" cl	Sweet	35Y–7.6R		35Y–3.5R	¼	
	Bl.P.S.Y.		" cl	Sweet	35Y–7.6R	35Y–3.5R	20Y–2.5R	¼	
Bl.P.S.Y.		Bl.P.S.Y.	" cl	Sweet	35Y–7.6R		20Y–2.5R	¼	
P. Winter Y.			" cl b	Sweet	35Y–7.6R			¼	Must pass
	P. Winter Y.		" cl b	Sweet	35Y–7.6R	35Y–3.5R		¼	Must pass
	Light Colored P.S.Y.		" cl	Sweet	35Y–3.5R	35Y–2.5R		¼	
P.S. White	P.S. White		" cl	Sweet	20Y–2.5R			¼	
P. Winter White	P. Winter White		" cl b	Sweet	20Y–2.5R			¼	Must pass
G.O.S.Y.			"	Off	35Y–7.6R			¼	
	G.O.S.Y.		" cl	Inferior	35Y–7.6R	35Y–3.5R		¼	
		G.O.S.Y.	" cl	Off	35Y–7.6R		20Y–3.5R	¼	
O.S.Y.			" cl	Off	35Y–12R			½	
	O.S.Y.			Inferior	35Y–12R			½	
Reddish O.S.Y.			"	Inferior	35Y–20R			¾	
	Reddish O.S.Y.			Inferior	35Y–20R			¾	

C —Choice
P —Prime
S —Summer
Y —Yellow
Bl—Bleachable

G —Good
O —Off
cl —Clear
b —Brilliant

drogenated oil of a comparatively low melting point and with only a small amount of highly unsaturated glycerides is desired.

On the other hand a cake baker when using hydrogenated oil in the baking of his cake is interested in receiving a hydrogenated oil which will cream well and which has high water-absorption qualities.

Hydrogenated oils naturally show entirely different chemical and physical characteristics from simply alkali-refined oil. In spite of this the hydrogenation process can, I believe, be justly considered as part of the refining process, eliminating or reducing the undesirable constituents of the oil and increasing or creating new desirable components.

Rancidity

Under certain conditions, and especially if refined cottonseed oil is exposed to the light, it will develop a rancid or unpleasant taste. What chemical constituents or what action causes the development of rancidity I believe has not been fully determined. Probably sometimes it is caused by the presence of ketones or aldehydes, and only very seldom is it directly related to the presence of free fatty acids.

It has been found that sometimes oil with a rancid taste will give the so-called Kreis test reaction, and the consumer not fully acquainted with the chemistry of oil has generalized this experience and thinks that all oils showing the Kreis test reaction must naturally be rancid. Such a conclusion is very unwarranted. Perfectly sweet oil, absolutely free from rancidity, may show the Kreis test reaction, and in many cases neither alkali refining nor deodorization nor hydrogenation will eliminate the Kreis test reaction.

I have tried to show with my arguments that from the manufacturer's or consumer's point of view the expression "Refined Oil" is a rather broad definition, and today probably means quite different qualities from those it represented twenty or forty years ago. This is due to the continuous development of the industry, for which we to a great extent have to thank the Chemist.

It is also to the Chemist that we look for new discoveries concerning the properties of cottonseed oil and for new methods of refining, so that the so-called refining loss, which is now actually an enormous economic waste, can be materially decreased, so that new qualities can be produced to increase the value to present users, and further, so that new channels for its use can be developed, thus creating a still greater consuming demand for the commodity.

Postum Buys Hellman

Cereal and Health Drink Line Augmented by Mayonnaise

THE Postum Company announces that it has made a contract to purchase the entire business of Richard Hellman, Inc., mayonnaise manufacturers.

Common stockholders of Hellman will receive one share of Postum stock for each 3.7 shares of Hellman stock.

Referee Applicants

APPLICANTS for Referee Chemist Certificates are Herbert M. Shilstone, New Orleans, (second publication), and Herman A. Nester, San Antonio Texas, (first publication).

Y—Yellow
Bl—Bleachable
b —Brilliant

Chemical Industries Exposition

Eleventh Annual Display Held in September Will Be International in Scope.

THE history of Chemistry and the Chemical Industries is replete with developments that have moved so swiftly that to the layman they seem to have come from nowhere. In the argot they Have Arrived. This is particularly so in Plastics, Lacquers and the metal sections. One of the outstanding features developed at the last meeting of the Electro-Chemical Society in Philadelphia was a controversy upon the gaseous processes of reduction of metals from their ores in which proponents from two important industries debated with keen interest what may eventually become a systematic procedure in the metallurgical industries. This discussion created wide interest and undoubtedly will lead to many interesting developments at the Eleventh Exposition of Chemical Industries to be held at the Grand Central Palace, New York City, from September 26th to October 1st, 1927.

For the first time Foreign Exhibitors will show the latest developments in foreign chemistry and already such interest is causing American industries to take notice of the competition from overseas.

The Students' Course under the direction of Professor W. T. Read, Head of the Chemistry Department at the Technological College, Lubbock, Texas, is already developing more interest than ever before. It is contemplated to gather together all lectures delivered during the Students' Course and to put them in book form after the exposition to be sold to anyone interested. Such work if carried out will gather as a collection in a book for reference what some of the foremost authorities of the day will have contributed as their bit toward making the most intense and remarkable Students' Course of the Chemical Industries Exposition.

Among other important functions held during the exposition will be the meetings of the American Ceramic Society collaborating with the Student's Course program and the meeting of the American Salesmen's Association of the American Chemical Industry and some dozen or more organizations joining in the Chemical Industries Banquet to be held at the Hotel Roosevelt the evening of September 28th. The committee in charge is arranging details which will be announced later.

In addition a great deal of interest is being shown in new developments in Grinding Mills and in the improvements all along the line of Chemical Engineering that will be shown at the exposition.

Today the world recognizes the value of Chemistry and Chemical Engineering as never before, as the following quotation from Dr. Arthur D. Little's pamphlet to Chemists, Engineers and Managers, of April 1927, will show. Ernest Elmo Colkins says, "The manufacturer, whatever he may make, however basic and staple, however well entrenched in the homes of the country, can no longer settle down and let things take their course. He must now hold himself ready to act and act quickly, interpret the signs, anticipate

the new attitude of the public, analyze each new invention or discovery for its effects, immediate or ultimate, on his own business."

The brief paragraph quoted expresses more fully than many volumes why the Chemical Industries are again going ahead. The day is past when any industry can rest on its laurels of past performances. Unless a vision of the future is with us in the present, achievements are as water over the dam. The Chemical Industry has begun to visualize, with the result that the forward march is on again. Quoting John E. Teeple in "The Indicator" of November 26th, 1926: "One of the great sources of difficulty between nations in the past has been the urge of some one nation to possess land because the land contained a specific raw material and that specific raw material was absolutely necessary to supply some need of the world. The farther the chemist progresses in his work the more fully will this source of friction be removed."

At no place in the world will there be such a gathering of industry. The authorities in Chemistry and Chemical Engineering will discuss problems and explain new developments both domestic and foreign at this year's exposition and with the interest that is already manifested by foreign manufacturers, American Industry must look to its laurels.

Sidebottom Joins Darco Staff

Well-known Chemical Engineer to Assist President of Carbon Sales Organization.

THE Darco Sales Corporation announces the appointment of Mr. Herbert G. Sidebottom as Assistant to the President, Mr. George C. Lewis.

Mr. Sidebottom has had a wide experience in the Chemical Industry. Graduating with the 1913 Class of Chemical Engineers at Cornell University, his activities in the chemical field since that time have been numerous. For many years he was Secretary of the New York Section of The American Chemical Society, and also Secretary of the Chemists' Club. He is a member of The American Institute of Chemical Engineers.

The Company is to be congratulated on this addition to its staff of Engineering Specialists.

Herbert G. Sidebottom

Abstracts from Other Journals

DAVID WESSON, *Abstract Editor*

Determination of the Iodine Value in Aqueous Emulsion. J. Fialkow. (*Z. anal. Chem.*, 1927, 70, 227-229.)—In the determination of the iodine value of a fat Margosches and Hinner (*Z. angew. Chem.*, 1924, 37, 202) add water to the mixture of the alcoholic solution of the fat and the iodine in order to further both hydrolysis of the iodine and formation of hypoiodous acid, and also to obtain a large surface of contact between the iodine solution and the fat by distributing the latter throughout the liquid in very fine drops. The author finds that these objects may be attained and the use of alcohol entirely avoided if the fat is emulsified by means of gum arabic. The fat (0.1 to 0.15 grm.) is rubbed in a small porcelain dish with about one-half of its weight of the powdered gum and 1 to 2 drops of water until a uniform emulsion is obtained. This is treated with 5 to 10 c.c. of water, which is added dropwise at first, the mass being constantly stirred during the addition. The turbid emulsion thus obtained is transferred to a flask with a ground stopper, the dish being carefully rinsed. The liquid is mixed with 20 c.c. of 0.2 *N* iodine solution (1 part I; 1 part KI) and then with sufficient water to make the total volume 200 to 250 c.c., and after 5 minutes the whole is titrated with 0.1 *N* thiosulphate solution. The titre of the iodine solution is determined by a blank experiment similarly carried out. This procedure gives results in agreement with those furnished by Hübl's method, but is not readily applicable to the solid fats which are difficult to emulsify.

Salts of Linolenic Hexabromide from Lumbang Oil. G. A. Imperial and A. P. West. (*Phil. J. Sc.*, 1926, 31, 441-449.) — Linolenic acid hexabromide was prepared in quantity from Philippine lumbang oil, which consists almost entirely of the glycerides of linolenic, linolic, and oleic acids. The prepared acid was analyzed by determining the bromine content, and salts were prepared from it by first converting the acid into the potassium salts, treating a methyl alcohol solution of the salt with an inorganic salt, such as barium bromide, and purifying the precipitated linolenic hexabromide salt. The barium, zinc and lead salts were thus prepared. The zinc salt gave the best melting point, decomposing sharply at 174° C. Linolenic hexabromide and its salts are not very soluble in ordinary organic solvents; about 10 grms. of the acid dissolves in 200 c.c. of benzyl alcohol at 100° C.; 5 grms. in 500 c.c. of warm isobutyl alcohol, and 21 grms. in 400 c.c. of warm xylene. The salts are still less soluble, but all are soluble (to the extent of 1 to 4 per cent.) in hot benzaldehyde and glacial acetic acid; the barium, zinc and lead salts in hot benzyl alcohol, the zinc salt also in nitrobenzene and pyridine, and the lead salt also in nitrobenzene, whilst the potassium salt is soluble in hot normal propyl alcohol.